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SPECIFIC-GRAVITY SEPARATION

APPLIED TO THE ANALYSIS OF MINING EXPLOSIVES

BY

C. G. STORM

AND

A. L. HYDE

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SPECIFIC-GRAVITY SEPARATION APPLIED TO THE ANALYSIS OF MINING EXPLOSIVES.

By C. G. Storm and A. L. Hyde.

INTRODUCTION.

In the course of its investigations to determine what explosives can be used with least danger and greatest efficiency in mining operations, the Bureau of Mines has analyzed many samples of explosives and has devised methods for increasing the accuracy and lessening the cost of analytical work. It has also examined various processes, among them those devised for separating the different solid ingredients of mixtures by differences of specific gravity.

Separation by specific-gravity methods is utilized in quantitative chemical work, but its most important application is probably in the mining industry, for concentrating ores and minerals. In such concentrating, as a rule, the specific gravity of the material to be recovered is quite different from that of the material to be thrown away, so that the separation involves no great difficulties. In other industrial applications, by the use of centrifugal force, advantage may be taken of much smaller differences in specific gravity, as, for example, in the well-known process of separating cream from milk.

USE OF LIQUIDS IN GRAVITY SEPARATIONS.

In mineralogy, liquids of different specific gravities are sometimes used for separating different minerals as well as for determining their specific gravities.

LIQUIDS USED BY VARIOUS INVESTIGATORS.

Sonstadt[a] used ethyl iodide (specific gravity approximately 2.0) diluted with CS_2 for exact determinations of the specific gravities of various salts. By using a series of such mixtures having known densities, and by obtaining intermediate values by slightly altering the temperature of the mixtures, he was able to cover a wide range of specific gravities. The specific gravity of a solution in which a

[a] Sonstadt, E., New method of taking specific gravities, adapted for special cases: Chem. News, vol. 29, 1874, p. 127.

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crystal would neither float nor sink, but remain suspended, was taken as the specific gravity of the crystal. Sonstadt also used a water solution of pure potassium iodide and mercuric chloride (specific gravity, about 3.085), which was diluted to any desired extent. He suggested the use of this liquid as a convenient means for testing the specific gravity of minerals; for example, for separating diamonds or diamond dust from quartz sand.

Thoulet [a] used a similar solution containing 539 parts HgI_2 and 435 parts KI to 100 parts water.

Klein [b] made use of a solution of cadmium tungsten borate, consisting of $9WO_3.B_2O_3.2CdO + 18H_2O$ (specific gravity, 3.281).

Rohrbach's [c] solution consisted of a water solution of barium mercuric iodide with a specific gravity of about 3.58.

Brauns [d] proposed the use of methylene iodide, CH_2I_2 (specific gravity, 3.33); by dilution with benzene, solutions of lower density may be obtained.

Biedermann [e] refers to the use of mercurous nitrate, fusing at 76° C. to a liquid with a specific gravity of 5.3, which may be diluted with water.

Muthman [f] used acetylene tetrabromide ($C_2H_2Br_4$) with a specific gravity of 2.95.

Retgers [g] discovered a double salt of silver and thallium ($AgNO_3.TlNO_3$) that fuses at 75° C. to a clear liquid with a specific gravity of 4.8 and is miscible with water. This solution must be used warm, as it solidifies on cooling. Separations of minerals may be made with it in an ordinary test tube.

For use in connection with Retgers's solution, Penfield [h] describes an apparatus consisting of a glass tube about 20 cm. long and with an inside diameter of 2.2 cm., the bottom of which was gradually tapered, terminating in an opening about one-fourth of an inch in diameter, the tapered part being ground on the outside to take a glass cap of suitable capacity and on the inside to receive a long hollow stopper the upper end of which projected beyond the top of the tube. The

[a] Thoulet, M. J., Séparation mécanique des divers éléments minéralogiques des roches, Bull. Soc. Min. France, vol. 2, 1879, pp. 17-24; Note sur un nouveau procédé pour prendre la densité de minéraux en fragments très petits, vol. 2, pp. 189-191.

[b] Klein, Daniel, Sur la séparation mécanique par voie humide des minéraux des densité inférieure à 3.6: Bull. Soc. Min. France, vol. 4, 1881, pp. 149-155.

[c] Goldschmidt, V., Ueber Verwendbarkeit einer Kaliumquecksilberjodidlosüng bei mineralogischen und petrographischen Untersuchungen: Jahrb. Min. Beil., vol. 1, 1881, p. 179.

[d] Brauns, R., Ueber die Verwendbarkeit des Methylenjodids bei petrographischen und optischen Untersuchungen: Jahrb. Min., No. 2, 1886, p. 72.

[e] Biedermann, ——., Chemischer Kalender, pt. 2, 1910, p. 337.

[f] Muthman, W., Ueber eine zur Trennung von Mineralgemischen geeignete schwere Flüssigkeit: Zeitschr. Krystal. Min., vol. 30, 1899, pp. 73, 74.

[g] Retgers, J. W., Ueber schwere Flüssigkeiten zur Trennung von Mineralien: Jahrb. Min., No. 2, 1889, pp. 185-192; Thalliumsilbernitrat als schwere Schmelze zu Mineraltrennungen: Jahrb. Min., No. 1, 1893, pp. 90-94.

[h] Penfield, S. L., On some devices for the separation of minerals of high specific gravity: Am. Jour. Sci., ser. 3, vol. 50, 1895, p. 446.

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mineral to be separated was treated in the tube, from which the stopper had been removed, allowing the heavy part to settle into the detachable bottom cap. After the stopper had been inserted into the bottom of the tube, the cap containing the heavy material was removed for examination. The tube with its contents was placed inside of a larger tube in order that the entire apparatus could be warmed in a suitable bath.

An apparatus similar to that described by Penfield was prepared by the writers of this paper and used in some of the tests described.

WORK OF FRIEDENTHAL.

Among more recent applications of gravity separation by liquids to the quantitative analysis of mixtures should be mentioned the work of Friedenthal.[a] By the use of bromoform and xylene, Friedenthal obtained liquid mixtures of any desired specific gravity from 0.7 to 2.83. A mixture of the two substances to be separated was added to a mixture of these liquids having a specific gravity intermediate between the specific gravities of the two solids. This mixture was centrifuged at a high velocity in a special form of separating tube provided with a wide-bore stopcock. With this apparatus Friedenthal separated quantitatively sodium chloride (specific gravity, 2.13) from potassium chloride (specific gravity, 1.99). By centrifuging the mixture at 10,000 revolutions per minute he separated casein quantitatively from milk in 3 hours. Under these conditions milk fats separate into a solid and a liquid layer. Saturated fats were separated from unsaturated fats by treatment with iodine and subsequent centrifuging.

WHEN CHEMICAL ANALYSIS FAILS.

A purely chemical analysis is entirely inadequate for many explosive mixtures, particularly coal-mining explosives. For example, if the water extract of an explosive is found to contain both a nitrate and a chloride combined with sodium and ammonium, it is impossible to ascertain by chemical analysis the exact manner of combination of the ions. Thus, a mixture of equimolecular proportions of $NaNO_3$, NH_4NO_3, NaCl and NH_4Cl will be found on analysis to contain 16.61 per cent Na, 44.76 per cent NO_3, 13 per cent NH_4, and 25.63 per cent Cl. The tabulation following shows a few of the innumerable combinations that on analysis would give the same results.

As each of these substances has its particular function in an explosive mixture, obviously a knowledge of the exact combination of salts contained in the explosive is of considerable importance. Combinations of salts showing the same composition on chemical analysis are listed below.

[a] Friedenthal, Hans, Über quantitative chemische Analyse von Gemengen mit Verwendung der Differenzen im spezifischen Gewicht: Ber. Deutsch. chem. Gesell., vol. 44, pt. 1, 1911, pp. 904–909.

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Combinations of salts that on chemical analysis show the same composition.

Combination No.	Constituents.	Molecular weight.	Percentage in combination.	Composition by analysis.			
				Na.	NO_3.	NH_4.	Cl.
				Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
1	$NaNO_3$	85	30.69	16.61	44.76	13.00	25.63
	NH_4NO_3	80	28.88				
	NaCl	58.5	21.12				
	NH_4Cl	53.5	19.31				
		277.0	100.00				
2	$2NaNO_3$	170	61.37	16.61	44.76	13.00	25.63
	$2NH_4Cl$	107	38.63				
		277	100.00				
3	$2NH_4NO_3$	160	57.76	16.61	44.76	13.00	25.63
	2NaCl	117	42.24				
		277	100.00				
4	$1\frac{1}{2}NaNO_3$	127.5	46.03	16.61	44.76	13.00	25.63
	$\frac{1}{2}$NaCl	29.25	10.56				
	$\frac{1}{2}NH_4NO_3$	40	14.44				
	$1\frac{1}{2}NH_4Cl$	80.25	28.97				
		277.00	100.00				

QUANTITATIVE RESULTS HARD TO OBTAIN.

One of the writers suggested the possibility of making a qualitative, if not a quantitative, analysis of such mixtures in explosives by the use of liquids of such specific gravities that certain of the components of the mixtures would sink and others float when mixed with these liquids, the liquids necessarily to be of such nature that they would neither dissolve nor react with the ingredients to be separated. However, in the separation of such salts from explosive mixtures, there are difficulties that prevent the obtaining of quantitative results. Most explosives to which the method might be applicable contain some carbonaceous constituent such as wood pulp, wheat flour, or corn meal, and it is practically impossible to bring a mixture of this character into such a condition that some of the particles of the salts will not cling to the fibers of the wood pulp or become entangled with the starch granules, etc. Even fine pulverization of the mixture does not suffice to prevent such entanglement.

However, the tests made have shown that the gravity method of separation is often of much value in giving a qualitative analysis showing what salts are present and in supplying a rough quantitative separation.

THE LIQUIDS MOST SUITABLE.

The choice of a liquid for use in such separations is a matter of some importance. Water solutions are not suitable because the salts to be separated are soluble in water. Among organic liquids, either methylene iodide, acetylene tetrabromide, or bromoform is

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suitable, the latter two being preferred because their lower boiling points cause them to be more readily driven off for an approximately quantitative separation.

Mixtures of bromoform (specific gravity, 2.83) and chloroform (specific gravity, 1.49) were found particularly suitable because the specific gravity of most of the salts used in blasting explosives lies between 1.50 and 2.50. As these two liquids are miscible in all proportions they offer a means of obtaining a liquid of any desired density between the limits of their respective specific gravities.

SPECIFIC GRAVITIES OF SALTS IN EXPLOSIVES.

The specific gravities of some of the more common salts occurring in blasting explosives are given below: [a]

Specific gravities of salts occurring in blasting explosives.

Salt.	Specific gravity.	Temperature.	Authority.
		°C.	
Ammonium nitrate	1.74	.....	Schröder.
Ammonium chloride	1.52	.....	Do.
Ammonium sulphate	1.77	20	Retgers.
Ammonium alum (crystals)	1.62	.....	Clarke, F. W.
Ammonium perchlorate	1.87	.....	Woulf.
Sodium nitrate	2.26	.....	Clarke, F. W.
Sodium chloride	2.17	17	Retgers.
Sodium sulphate+$10H_2O$	1.46	.....	Clarke, F. W.
Sodium sulphate (anhydrous)	2.66	.....	Do.
Potassium nitrate	2.09	.....	Do.
Potassium chloride	1.99	16	Retgers.
Potassium alum (crystals)	1.75	17	Do.
Potassium sulphate	2.66	20	Tutton.
Magnesium sulphate+$7H_2O$	1.68	.....	Clarke, F. W.
Magnesium sulphate (anhydrous)	2.65	.....	Do.
Potassium chlorate	2.33	.....	Do.
Potassium perchlorate	2.52	.....	Schröder.
Calcium carbonate (precipitated)	2.72	.....	Rose, G.
Calcium sulphate (anhydrous)	2.97	.....	Schröder.
Calcium sulphate+$2H_2O$	2.32	.....	Clarke, F. W.
Barium nitrate	3.23	.....	Do.
Magnesium carbonate	3.04	.....	Schröder.
Manganese dioxide	5.03	.....	Retgers.

PROPER SPECIFIC GRAVITY OF TESTING LIQUID.

The specific gravity of the liquid to be used in separating any mixture of salts will, of course, depend on the specific gravities of the salts in the mixture. Manifestly, if a mixture of ammonium nitrate, sodium nitrate, and sodium chloride is stirred into a liquid with a specific gravity of about 2.00, the sodium salts will tend to settle to the bottom, whereas the ammonium salt will float.

METHOD OF PROCEDURE.

The following method of procedure has been found to be satisfactory:

The explosive to be examined is first extracted with ether in order to remove nitroglycerin, oils, or other materials of similar nature,

[a] Landolt and Börnstein, Physikalisch-chemische Tabellen, 1905, pp. 230-251.

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that tend to cause the crystalline particles to adhere to each other. The residue is dried in an oven and then ground finely in a mortar. The grinding is performed as rapidly as possible in order that absorption of moisture may be avoided. About 10 grams of this pulverized material is then added to about 25 c. c. of the mixture of bromoform and chloroform in a separatory funnel of about 100-c. c. capacity, the cock of which has an unusually wide bore. More of the liquid is added until the total volume is 50 to 75 c. c. The mixture is then well stirred with a glass rod and allowed to stand for a short time. This process causes the solid material to be divided into a heavy layer at the bottom, and a light layer floating on the surface of the liquid. The upper layer includes the wood pulp and other carbonaceous material together with the lighter salts.

The heavier salt may be drawn off into a dish or beaker by quickly opening the cock and again closing it before the lighter solids begin to come through. The greater part of the liquid may be decanted back into the separatory funnel and the small amount adhering to the crystals may be removed by evaporation, the heavier crystals being left in condition to be examined chemically.

The liquid in the funnel may be filtered from the mixture of lighter solids and used repeatedly for other tests, its specific gravity being determined occasionally in order that it may not vary too much from the desired density.

It may be found possible to separate a part of the lighter salt from the carbonaceous material by further treatment in a similar manner with a liquid of less density than the one first used.

The separation is in no case complete because particles of the different salts, especially if not thoroughly dry, have a tendency to cling together, forming composite particles whose specific gravity is between the specific gravities of the separate salts. Frequently such composite particles have about the same specific gravity as that of the separating liquid, and thus prevent a sharp separation. As has previously been mentioned, particles of the carbonaceous material also adhere to the crystals of both the light and the heavy salts, and, finally, air bubbles attached to some of the particles make their apparent specific gravities lower than their real specific gravities.

In spite of these adverse influences there is usually sufficiently complete separation to enable one to ascertain correctly the kinds and the amounts of the components of the mixture when the ultimate chemical composition of the mixture is known.

APPARATUS USED.

A simple separating tube with detachable bottom cap and inner stopper, similar to the apparatus of Penfield, described above, was used in some of the experiments of the writers, and proved, in some

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cases, to be more convenient than the ordinary separating funnel with wide-bore stopcock. Its construction is simple and its use avoids any clogging of the bore in the stopcock, as the opening in the bottom of the tube may be made as wide as desired.

EXPERIMENTS WITH PURE SALTS.

A few experiments were performed with pure salts in order to determine the density of liquid required to give the most practical separations of the different salts involved. Below is noted the behavior of a few salts when placed in liquids with specific gravities slightly greater or less than those of the salts:

$NaNO_3$ (specific gravity, 2.26) sinks in a liquid with a specific gravity of 2.16.

NaCl (specific gravity, 2.17) nearly all floats in a liquid with a specific gravity of 2.16.

NaCl (specific gravity, 2.17) sinks in a liquid with a specific gravity of 1.98.

KNO^3 (specific gravity, 2.09) nearly all floats in a liquid with a specific gravity of 2.16.

KNO_3 (specific gravity, 2.09) sinks in a liquid with a specific gravity of 1.98.

NH_4NO_3 (specific gravity, 1.74) floats in a liquid with a specific gravity of 1.77.

NH_4NO_3 (specific gravity, 1.74) sinks in a liquid with a specific gravity of 1.60.

NH_4Cl (specific gravity, 1.52) floats in a liquid with a specific gravity of 1.60.

$Na_2SO_4.10H_2O$ (specific gravity, 1.46) nearly all floats in a liquid with a specific gravity of 1.60.

A difference of about 0.1 between the specific gravity of the liquid and that of the salt is necessary in order that the salt shall either float or sink with any degree of definiteness. It must be remembered that in separating a salt from an explosive mixture, its apparent specific gravity will be somewhat less than its real specific gravity on account of the buoyant effect of air bubbles, particles of wood pulp or other carbonaceous material, etc., so that in selecting a liquid for the separation of two salts it is better to have the specific gravity of the liquid nearer to that of the lighter salt than to that of the heavier. Thus, for separating NaCl from NH_4NO_3 it would be better to have the specific gravity of the liquid nearer to 1.71 than to 2.13.

EXPERIMENTS WITH MIXTURES OF SALTS.

Several mixtures of salts were prepared in proportions such as are liable to occur in explosives, and separations were made with mixtures of chloroform and bromoform.

Mixture 1 consisted of 0.5 gram of NH_4Cl, 0.5 gram of $NaNO_3$, 0.5 gram of wood pulp, and 8.5 grams of NH_4NO_3. This mixture was treated with a liquid with a specific gravity of 1.89. The part that sank to the bottom weighed 0.422 gram and was practically pure $NaNO_3$, showing only traces of NH_3 or Cl. The part that floated contained practically all the ammonium salts and a small quantity of the sodium salt.

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Mixture 2 consisted of 0.5 gram of NaCl, 0.5 gram of $NaNO_3$, 0.5 gram of wood pulp, and 8.5 grams of NH_4NO_3. This was treated with a liquid with a specific gravity of 1.89. The part that sank to the bottom weighed 0.85 gram and consisted of a mixture of practically pure NaCl and $NaNO_3$; it showed only a trace of NH_3. The part that floated contained practically all the NH_4NO_3 with some NaCl and $NaNO_3$.

Mixture 3 had the same composition as mixture 2. It was treated with a liquid with a specific gravity of 1.77. The part that sank weighed 0.87 gram. Qualitative tests of different parts showed the same results as for mixture 2.

Mixture 4 consisted of 0.5 gram of NH_4Cl, 0.5 gram of NaCl, 0.5 gram of wood pulp, and 8.5 grams of NH_4NO_3. The mixture was treated with a liquid with a specific gravity of 1.77. The part that sank weighed 0.364 gram and consisted of NaCl with only traces of ammonium salts. The part that floated contained practically all the ammonium salts.

Mixture 5 consisted of 0.5 gram of NH_4Cl, 0.5 gram of NaCl, 0.5 gram of $NaNO_3$, 0.5 gram of wood pulp, and 8 grams of NH_4NO_3. The mixture was treated with a liquid with a specific gravity of 1.77. The part that sank weighed 0.814 gram and consisted of $NaNO_3$ and NaCl with only traces of ammonium salts. The part that floated contained practically all the ammonium salts. This light part was separated and again treated in a small beaker with a liquid with a specific gravity of 1.60. The floating salt was skimmed off and gave qualitative tests for NH_4Cl.

Provided that the above mixtures had already been analyzed chemically so that the percentages of NH_4, Cl, NO_3, and Na were known, the qualitative analyses indicated were sufficient to show their real composition except in the last mixture. This mixture contained all four salts, and it is difficult to see how anything but a rough approximation to its real composition can be obtained.

EXPERIMENTS WITH TWO EXPLOSIVES.

The application of the method may be illustrated by its use with two actual explosives.

Explosive 1 was an ammonium nitrate explosive containing about 10 per cent of nitroglycerin. The water solution was found to contain NH_4, Na, NO_3, and Cl. Ordinary chemical analysis failed to show whether the sodium was present as a nitrate or a chloride. When the explosive was treated with a liquid with a specific gravity of about 1.8, crystals of $NaNO_3$ sank to the bottom, but no test for Cl was given by this heavier part of the mixture. The floating part of the mixture gave strong tests for NH_4, Cl, and NO_3. The mixture was therefore believed to consist of NH_4NO_3, $NaNO_3$, and NH_4Cl.

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Explosive 2 was an ammonium nitrate explosive containing about 10 per cent of nitroglycerin. The chemical analysis gave results very similar to those obtained with explosive 1, the water solution containing NH_4, Na, NO_3, and Cl. However, when treated with a liquid with a specific gravity of about 1.6, the part that sank showed no trace of NO_3, but was found to be nearly all NaCl. It was therefore concluded that the water-soluble material consisted of a mixture of NH_4NO_3 and NaCl.

CONCLUSION.

These illustrations are sufficient to show the possibilities as well as the limitations of the specific-gravity method. The test is easily and quickly made, and its indications seem to be sufficiently definite to make it of some value in the rather limited field in which it can be used.

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PUBLICATIONS ON TESTS OF EXPLOSIVES.

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BULLETIN 10. The use of permissible explosives, by J. J. Rutledge and Clarence Hall. 1912. 34 pp., 5 pls., 4 figs.

BULLETIN 15. Investigations of explosives used in coal mines, by Clarence Hall, W. O. Snelling, and S. P. Howell, with a chapter on the natural gas used at Pittsburgh, by G. A. Burrell, and an introduction by C. E. Munroe. 1911. 197 pp., 7 pls., 5 figs.

BULLETIN 17. A primer on explosives for coal miners, by C. E. Munroe and Clarence Hall. 61 pp., 10 pls., 12 figs. Reprint of United States Geological Survey Bulletin 423.

BULLETIN 48. The selection of explosives used in engineering and mining operations, by Clarence Hall and S. P. Howell. 1913. 50 pp., 3 pls., 7 figs.

BULLETIN 51. The analysis of black powder and dynamite, by W. O. Snelling and C. G. Storm. 1913. 80 pp., 5 pls., 5 figs.

BULLETIN 59. Investigations of detonators and electric detonators, by Clarence Hall and S. P. Howell. 1913. 73 pp., 7 pls., 5 figs.

BULLETIN 66. Tests of permissible explosives, by Clarence Hall and S. P. Howell. 1913. 313 pp., 1 pl., 6 figs.

TECHNICAL PAPER 6. The rate of burning of fuse as influenced by temperature and pressure, by W. O. Snelling and W. C. Cope. 1912. 28 pp.

TECHNICAL PAPER 7. Investigations of fuse and miners' squibs, by Clarence Hall and S. P. Howell. 1912. 19 pp.

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TECHNICAL PAPER 52. Permissible explosives tested prior to March 1, 1913, by Clarence Hall. 1913. 11 pp.

TECHNICAL PAPER 69. Production of explosives in the United States during the calendar year 1912, compiled by A. H. Fay. 1914. 8 pp.

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